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Voronoi cells, fractal dimensions and fibre composites

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Abstract

The use of fibre-reinforced polymer matrix composite materials is growing at a faster rate than GDP in many countries. An improved understanding of their processing and mechanical behaviour would extend the potential applications of these materials. For unidirectional composites, it is predicted that localised absence of fibres is related to longitudinal compression failure. The use of woven reinforcements permits more effective manufacture than for unidirectional fibres. It has been demonstrated experimentally that compression strengths of woven composites are reduced when fibres are clustered. Summerscales predicted that clustering of fibres would increase the permeability of the reinforcement and hence expedite the processing of these materials. Commercial fabrics are available which employ this concept using flow-enhancing bound tows. The net effect of clustering fibres is to enhance processability whilst reducing the mechanical properties.

The effects reported above were qualitative correlations. To improve the design tools for reinforcement fabrics we have sought to quantify the changes in the micro-/meso-structure of woven reinforcement fabrics. Gross differences in the appearance of laminate sections are apparent for different weave styles. The use of automated image analysis is essential for the quantification of subtle changes in fabric architecture. This paper considers Voronoi tessellation and fractal dimensions for the quantification of the microstructures of woven fibre-reinforced composites. It reviews our studies in the last decade of the process-property-structure relationships for commercial and experimental fabric reinforcements in an attempt to resolve the processing *versus* properties dilemma. A new flow-enhancement concept has been developed which has a reduced impact on laminate mechanical properties.

Introduction

The manufacture of fibre reinforced composites has been reviewed by Åström (1997) [1], Gutowski (1997) [2] and Davé and Loos (1999) [3]. Resin Transfer Moulding (RTM) (van Harten, 1993. Potter, 1997. Rudd *et al.*, 1997. Kruckenberg and Paton, 1998. Benjamin and Beckwith, 1999) [4-8] is emerging as the most probable route to mass production for composite components of complex shape. In RTM a mould is loaded with dry fibres, resin then flows into the dry fabric stack and the resin cures to produce a solid component. The success of the process is critically dependent on the rate at which the resin percolates through the fibres. The Darcy (1856) equation [9] is commonly used for simulation of the process. For a fixed geometry, the flow rate is proportional to the pressure gradient and inversely proportional to the resin viscosity. The constant of proportionality is known as the permeability of the porous medium. Summerscales (1993b) [10] predicted that clustering of fibres would increase the resin flow rate in the reinforcement and hence expedite the processing of these materials. Thirion *et al.* (1988) [11] have reported commercial fabrics which employ this concept using flow-enhancing bound tows.

Unidirectional (UD) fibres offer the highest mechanical performance when stresses are primarily in either tension or compression along the fibre axis. For more uniformly distributed stresses, it is common to use cross/angle-ply UD fibre composites. However, the absence of transverse reinforcement within each layer makes these materials liable to splitting parallel to the fibres. For applications where high stiffness and high strength are required together with toughness, woven composites can provide a reasonable balance of stiffness, strength and toughness whilst offering improved processability.

For unidirectional composites, the finite element method (FEM) has been used to predict that the type of packing (Wisnom, 1990) [12] or the degree of randomness (Guild *et al.*, 1990) [13] affects the transverse modulus. Further, FEM has indicated that localised absence of fibres is related to longitudinal compression failure (Guild *et al.*, 1989) [14]. Basford *et al.* (1995) [15] have demonstrated experimentally that compression strengths of woven composites were reduced when fibres were more clustered. The net effect of clustering fibres is generally to enhance processability whilst reducing the mechanical properties.

The effects reported above were qualitative correlations. To improve the design tools for reinforcement fabrics we have sought to quantify the changes in the micro-/meso-structure of woven reinforcement fabrics. Gross differences in the appearance of materialographic sections are apparent for different weave styles. For subtle variations within a single weave style, the eye cannot easily discern changes. The use of automated image analysis (Guild and Summerscales, 1993. Summerscales, 1998) [16, 17] is essential for the quantification of subtle changes in fabric architecture. The microstructure of fibre reinforced composites is normally defined by specifying the form of the reinforcement and

quantified by measuring the fibre volume fraction and the fibre length/orientation distributions. This data may be insufficient where clustering of fibres occurs. The classification of structured populations can be achieved by a variety of parameters. Early techniques included nearest-neighbour analysis (Clark and Evans, 1954) [18], chi-squared analysis for point patterns (Davis, 1974) [19], quadrat analysis (Greig-Smith, 1952) [20], mean free path and mean random spacing (Cribb, 1978) [21], space auto-correlograms (Mirza, 1970) [22], area fraction variance analysis and mean intercept length analysis (Li *et al.*, 1992) [23] and (for hybrid composites) contiguity index (Short and Summerscales, 1984) [24]. More recently the classification of the structures within composite materials has used either tessellation techniques (Summerscales *et al.*, 1993a. Pyrz, 1994, Ghosh *et al.*, 1997. Pyrz, 2000a. Pyrz, 2000b) [25-29] or fractal dimensions (Taya *et al.*, 1991. Cross, 1994. Worrall and Wells, 1996) [30-32].

This paper will consider Voronoi tessellation and fractal dimensions for quantification of the microstructures of woven fibre-reinforced composites and will review the process-property-structure relationships for commercial and experimental fabric reinforcement materials studied experimentally in an attempt to resolve the processing *versus* properties dilemma.

EXPERIMENTAL

Materials

Three sets of woven carbon fibre reinforcement fabrics were studied in the following temporal sequence:

- Carr Reinforcements twill fabric with bound Flow-Enhancing Tows (FET)
- Brochier twill, satin and Injectex FET satin
- Carr Reinforcements twill fabric with new concept FET

Carr fabrics with bound FET

The Carr fabrics with bound FET were based on a 380 g/m² 6K carbon 2x2 twill fabric (Figure 1). The flow enhancement was achieved by binding regularly spaced weft tows to constrain these tows to remain approximately elliptical under compression, thereby creating large porespaces adjacent to the FET. There were four variants plus the reference base fabric:

- fabric twill = normal
- fabric 156 = 12½% FET (1 in 8)
- fabric 150 = 17% FET (1 in 6)
- fabric 148 = 25% FET (1 in 4)
- fabric 126 = 50% FET (1 in 2)

The permeating fluids were Scott Bader Resin E (initial ambient viscosity (IAV): 4600 mPa.s [1 mPa.s = 1 centipoise]) and Jotun 4210 (IAV = 2600 mPa.s) unsaturated polyester resins. The results have been reported more fully elsewhere (Griffin *et al.*, 1995a/b. Guild *et al.*, 1996. Summerscales *et al.*, 1995) [33-36].

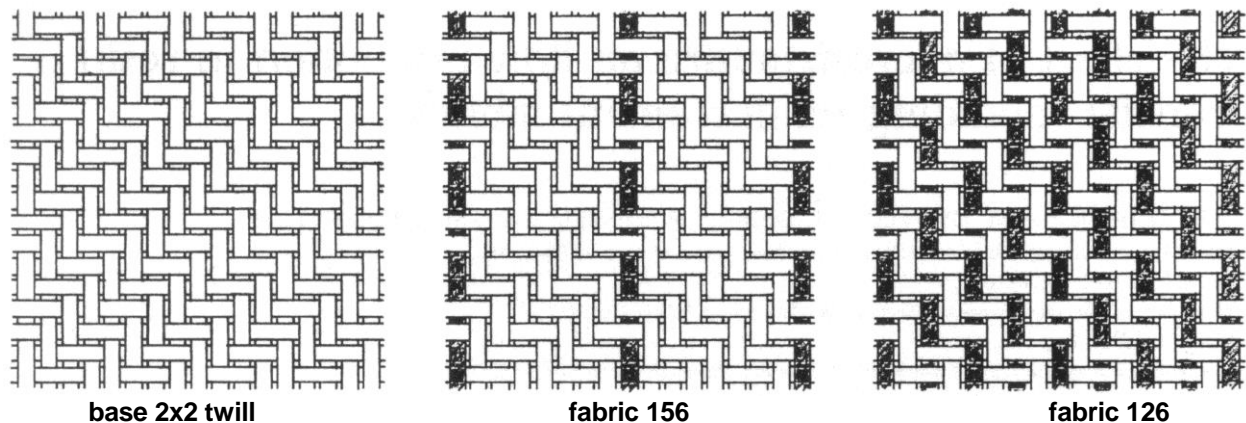


Figure 1: Schematics of Carr fabrics

Brochier fabrics

The Brochier fabrics were all 290 g/m² 6K carbon fibre fabrics (Figure 2):

- 2x2 twill (designated E3853/G986)
- normal 5-harness satin (designated E3833/G963)
- flow-enhanced Injectex 5-harness satin (every fifth tow bound in one direction, designated E3795)

The permeating fluid was Ciba-Geigy LY564-1/HY2954 epoxy resin (IAV ~600 mPa.s). The results have been reported more fully elsewhere (Pearce *et al.*, 1998a/b) [37, 38].

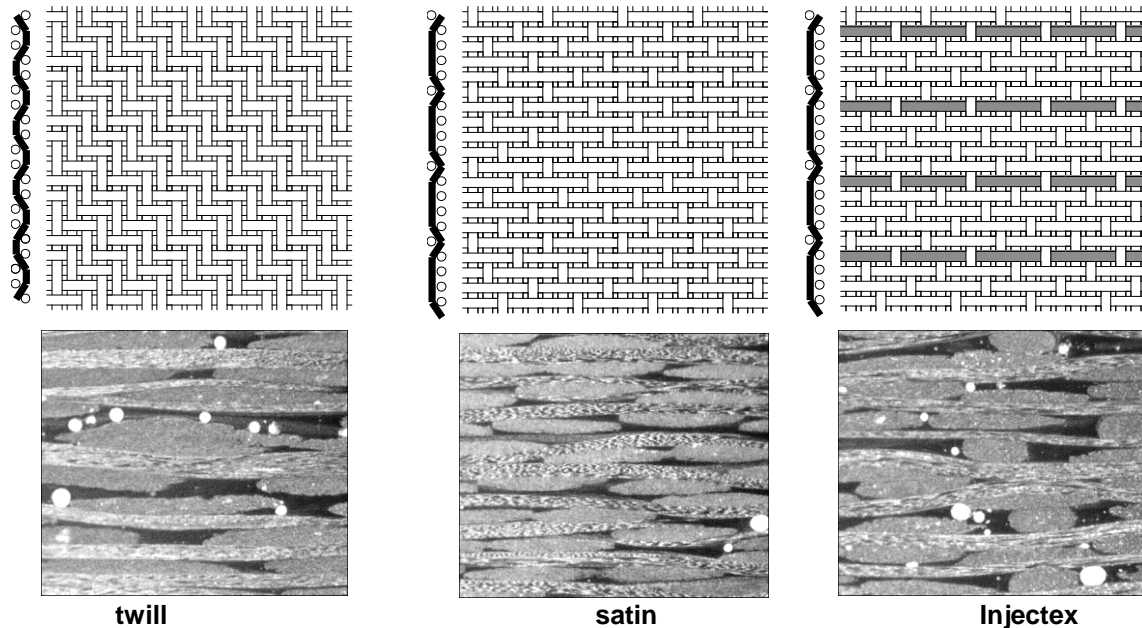


Figure 2: Schematics and transverse micrographs of Brochier weaves. Image frame is 3.6 x 3.0 mm.

New concept Carr fabrics

The new concept Carr fabrics are based on a 372 g/m² 6K carbon 2x2 twill fabric. Flow enhancement is achieved by substitution of some 6K weft tows by 3K tows with a consequent reduction in fabric areal weight:

| Fabric | Description | Areal weight |
|--------|------------------|----------------------|
| D | normal twill | 372 g/m ² |
| C | 14% FET (1 in 6) | 358 g/m ² |
| B | 20% FET (1 in 4) | 353 g/m ² |
| A | 33% FET (1 in 2) | 340 g/m ² |

It is not possible to discern differences between these fabrics with the unaided eye either for dry fabric or sectioned composites. The permeating fluid was SP Ampreg 26 epoxy resin with slow hardener (IAV = 310 mPa.s). The results have been reported more fully elsewhere (Pearce *et al.*, 2000) [39].

Measurement of permeability/fabrication of test plates

The composite plates were manufactured during radial flow permeability experiments conducted in a glass topped aluminium mould with controlled cavity depth. The apparatus and technique, as used for the Brochier fabrics, has been described elsewhere (Carter *et al.*, 1996) [40] and is subject to progressive refinement. Figure 3 shows frame grabbed images of the advancing flow front during a typical determination of permeability. The images were taken at 36, 201 and 653 seconds into mould fill. Carter *et al.* (1995) [41] have published the theory used for calculation of the permeability.

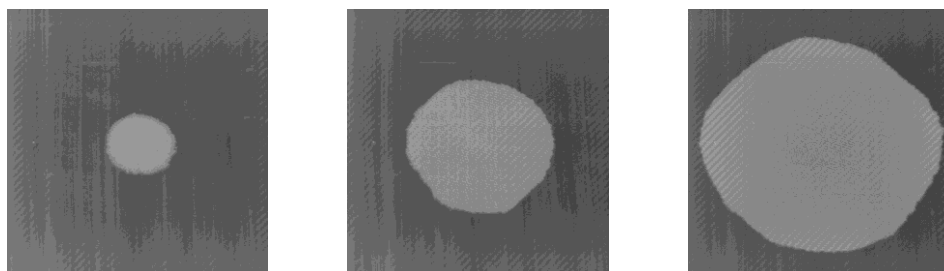


Figure 3: Frame-grabbed images of a typical radial flow permeability experiment at 36, 201 and 653 seconds. The square mould has an edge length of 440 mm.

Microscopical analysis

Preparation

The woven fabric composites were sampled in both warp and weft directions. Individual pieces of full specimen thickness by up to 25 mm length were mounted by casting in cylindrical pots. Each block was polished in six stages to 1 μm and finished with AlSiO_2 for 2 min (Pearce *et al.*, 1998a/b, Pearce *et al.*, 2000) [37-39].

Image analysis

Image analysis was undertaken using a Quantimet 570 image analysis system. The following features were measured: individual void areas, numbers of voids, total void area, counts and areas of porespace.

Dirichlet tessellations are constructed such that any point within a cell is closer to the centre of gravity of the feature within that cell than the centre of gravity of any other cell. The boundaries between Dirichlet cells are straight lines which are in effect the perpendicular bisectors between the centres of gravity of features. Dirichlet tessellation characterises clustering from the positions of the features

We have used a specific form of tessellation known as the Voronoi cell to characterise the clustering of the features. The features are "grown" until the surfaces meet and fill the whole space. This normally results in non-linear boundaries between cells. Characterisation of clustering by growing techniques is strongly influenced by the size and shape of the features as well as their positions. A rigorous treatment of the spatial statistics is given in Cressie (1993) [42]. Figure 4a illustrates how, for individual filaments within a bundle, each point in space is assigned to the nearest particle and Figure 4b shows the boundaries constructed from this information. The cells of the tessellation are then analysed to characterise the regions influenced by the non-overlapping particles. The parameters recorded were area, maximum width (horizontal feret), maximum height (vertical feret), perimeter and x - and y - centres of gravity.

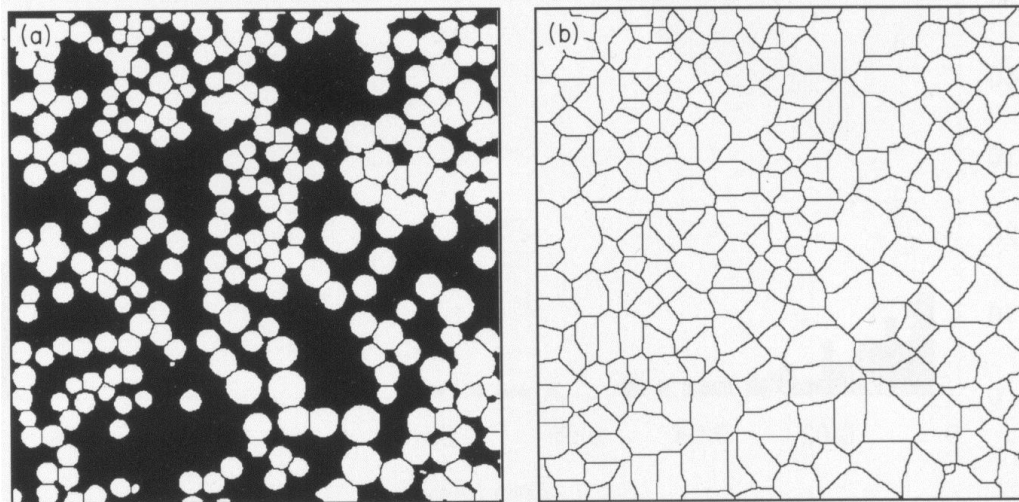


Figure 4: Voronoi tessellation of a section of glass fibre composite (fibre diameters $\sim 10\text{-}30\ \mu\text{m}$)

To determine the fractal dimension, the microscope image is recorded with 256 grey levels. For example, in Figure 5 we use a double threshold. Surface breaking voids are filled with talc and appear white to permit them to be quantified separately. They are then converted to black along with all the other (darker) intertow porespace by careful selection of threshold levels. The tows are then represented by white. The whole image is mapped with a grid of boxes and those boxes containing any porespace are flagged (Figure 5). A range of square box sizes is used. In each case, the porespace area is measured as the total 'flagged' area. The log (area of filled boxes) is plotted against log (box size). This is known as a Richardson plot. The slope of the graph is the fractal dimension (δ) and gives a unique measurement of the porespace size and distribution. The raw value returned by this process is reported here (note that this value is 0 for a line and 1 for an area. For more conventional representations of dimensions it is necessary to increase this number by unity). The measurement details are summarised in Table 1.

Table 1: Details of image analysis for determination of fractal dimensions

| | Brochier [37] | Brochier [38] | Carr new concept [39] |
|------------------------------|------------------------------|------------------------------|-----------------------------|
| Published reference | Pearce <i>et al.</i> , 1998a | Pearce <i>et al.</i> , 1998b | Pearce <i>et al.</i> , 2000 |
| Pixel frame | 512 x 400 | 480 x 400 | 480 x 480 |
| Analysis frame area (mm) | 4 x 3.125 | 3.6 x 3.0 | 2.2 x 2.2 |
| Linear resolution (nm/pixel) | 7812 | 7500 | 4583 |
| Contiguous frames per sample | six | three | four |
| Measured area (mm) | 24 x 3.125 | 10.8 x 3 | 8.8 x 2.2 |
| Specimens per fabric | one | ten | three |
| Total area per fabric | 75 mm ² /fabric | 324 mm ² /fabric | 58 mm ² /fabric |

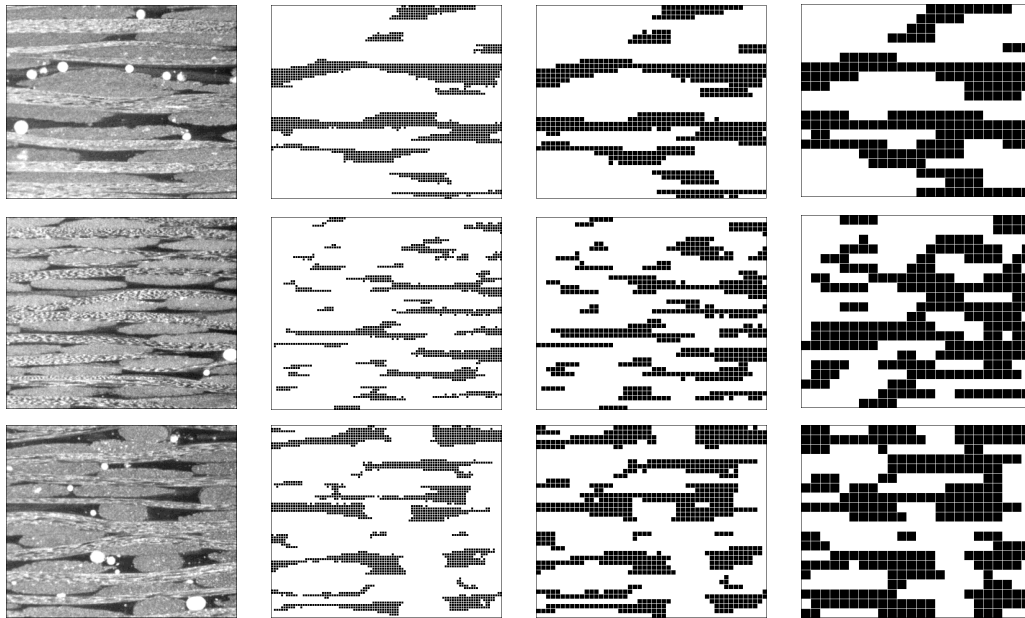


Figure 5: Sections of Brochier weaves with sample frames from the determination of fractal dimensions.

Top row: twill, middle row: satin, bottom row: Injectex. Image frame is 3.6 x 3.0 mm.

column 1: 480x400 pixel grey image, column 2: 4x4 pixel boxes, column 3: 9x9 pixel boxes, column 4: 19x19 pixel boxes

Mechanical testing

Mechanical testing was conducted using CRAG test methods (Curtis, 1988) [43]:

- Tensile properties were measured using CRAG method 302 at 5 mm/min crosshead displacement in an Instron 1175 screw-driven universal testing machine with a 100 kN load cell.
- Compression properties were measured using CRAG method 401 at an actuator displacement speed of 2.4 mm/min in an Instron 8500 servo-hydraulic universal testing machine with a 200 kN load cell.

except where otherwise stated (*i.e.* Carr bound FET). All specimens were monitored with TMG 350 Ω 12.5 mm strain gauges fed to a Strawberry Tree data logger recording at 10 Hz. Secant moduli were calculated at 2500 $\mu\epsilon$.

RESULTS

Carr bound tow FET fabrics

Initial attempts to quantify the microstructure of woven fabrics were conducted on Carr fabrics with bound FET. The areas of pore space were collected and histograms were drawn: for example, fabric 126 data is shown in figure 6a. Comparing the histograms, it was apparent that different shapes of histograms were associated with the different fabrics. The differences in the data is clearest when the cumulative curves are plotted as the number of zones *less* than a given size. These cumulative curves, for all 5 fabrics, are shown in figure 6b. The cumulative number has been 'normalised' to account for the small differences in total area measured for the different fabrics. This plot is truncated at small porespace areas corresponding to ~2% of the area of a typical tow. The relative separation of the plots at around 0.05 mm² appears to correlate with the relative values of permeability (Figure 7), albeit that the range of flow areas chosen is somewhat arbitrary.

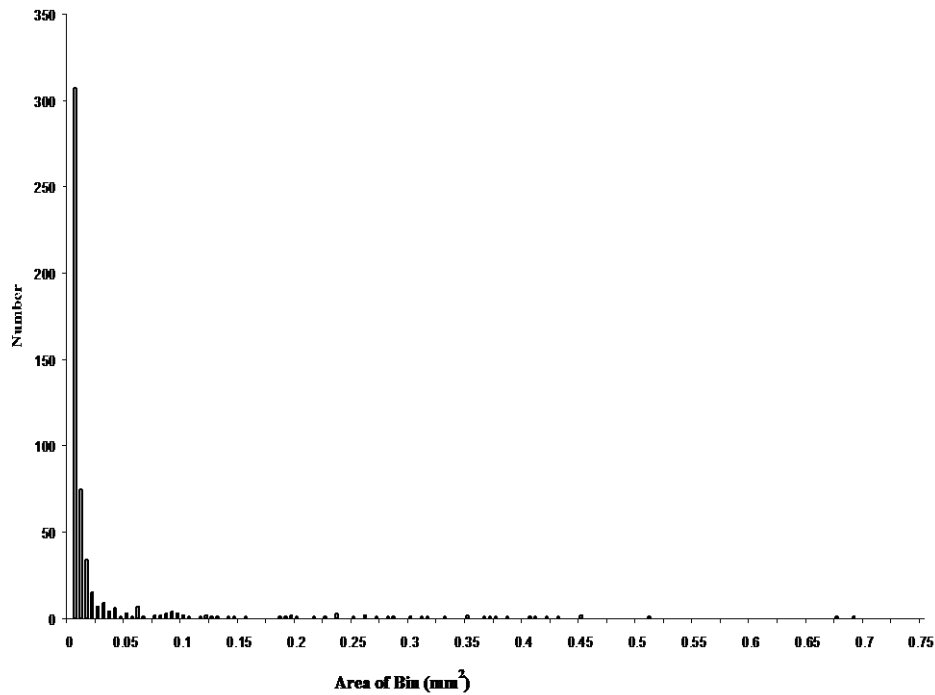


Figure 6a: Histogram of pore space areas for Carr fabric 126

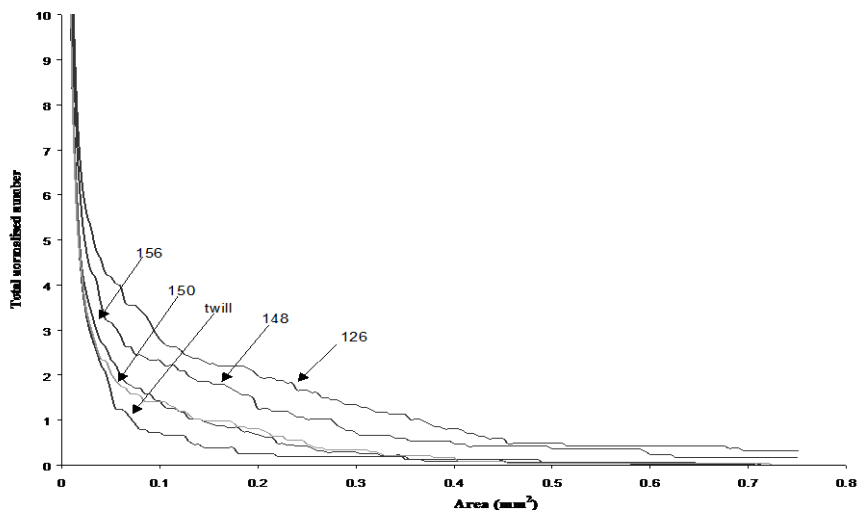


Figure 6b: Cumulative curve of pore space zones less than a given value for Carr fabrics



Figure 7: Flow area and permeability plotted against percentage FET for Carr bound tow FET

Basford *et al.* (1995) [15] measured mechanical properties for these composites at constant fibre volume fraction in the Instron 1175 screw-driven test machine. Both the compression strengths (CRAG method 401 at 2 mm/min) and the apparent interlaminar shear strengths (CRAG method 100 at 1 mm/min) decreased with increasing proportion of flow-enhancing tows at constant fibre volume fraction (~43%). The mean values for six specimens of each type are shown in Figure 8. The maximum standard deviation was 26 MPa for compression strengths and 5.2 MPa for ILSS.

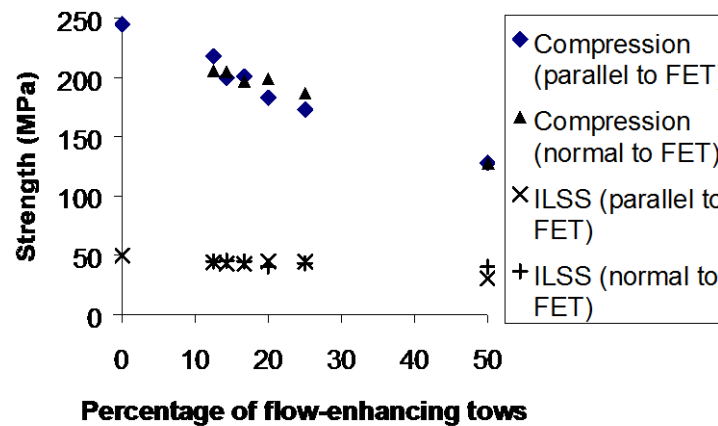


Figure 8: Strengths of Carr fabrics plotted against proportion of flow-enhancing tows in the weft.

Brochier fabrics

The use of fractal dimensions to analyse the porespace features normal to the flow enhancing tows clearly differentiates between the structures of the Brochier fabrics: Injectex FET ($\delta = 0.356$), twill ($\delta = 0.364$) and satin without FET ($\delta = 0.424$). The weave style influences porespace distribution (and hence permeability) and fibre crimp (and hence mechanical properties). The relative permeabilities of the fabrics are shown in Figure 9 and the mechanical properties are presented in Table 2. The ranked experimental results (Table 3) for the Brochier fabrics clearly show that a conflict exists between processing (satin is the worst fabric) and mechanical properties (satin is the best fabric).

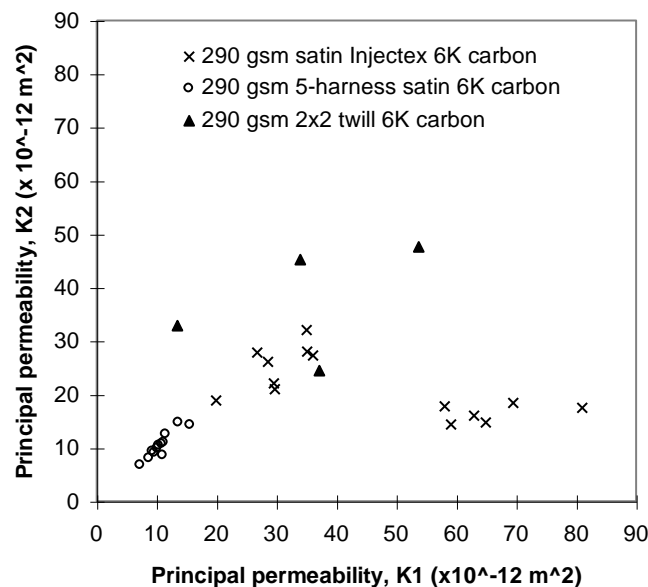


Figure 9: Principal in-plane permeabilities in x (K_1) and y (K_2) of the Brochier fabrics (units of permeability are $\times 10^{-12} \text{ m}^2$)

Table 2: Mechanical properties of Brochier fabrics normal to the FET

| | Compression | Tension |
|-----------------------------|----------------|----------------|
| Young's moduli (GPa) | | |
| Injectex | 50 ± 1.0 | 52 ± 2.4 |
| Satin | 54 ± 1.1 | 57 ± 1.3 |
| Twill | 51 ± 1.3 | 54 ± 1.3 |
| Strengths (MPa) | | |
| Injectex | 339 ± 37.2 | 767 ± 42.7 |
| Satin | 458 ± 66.2 | 906 ± 34.3 |
| Twill | 360 ± 56.5 | 781 ± 46.2 |

Table 3: Ranking of measured properties and quantified microstructures normal to the FET

| | Lowest | Middle | Highest |
|--------------------------------|----------|----------|----------|
| <i>Processing</i> | | | |
| Permeability | satin | Injectex | twill |
| <i>Mechanical properties</i> | | | |
| Tensile Strength/Moduli | Injectex | twill | satin |
| Compressive Strength/Moduli | Injectex | twill | satin |
| <i>Fractal analysis</i> | | | |
| Inter-tow porespace area | satin | twill | Injectex |
| Fractal dimension (δ) | Injectex | twill | satin |

New concept Carr fabrics

For the new concept Carr fabrics, the permeabilities and fractal dimensions are both ranked in the sequence ACBD (Figure 10). The fractal dimension (δ) of the base fabric (D) is essentially the same in warp and weft. The value of δ is similar for the warp direction for all fabrics.

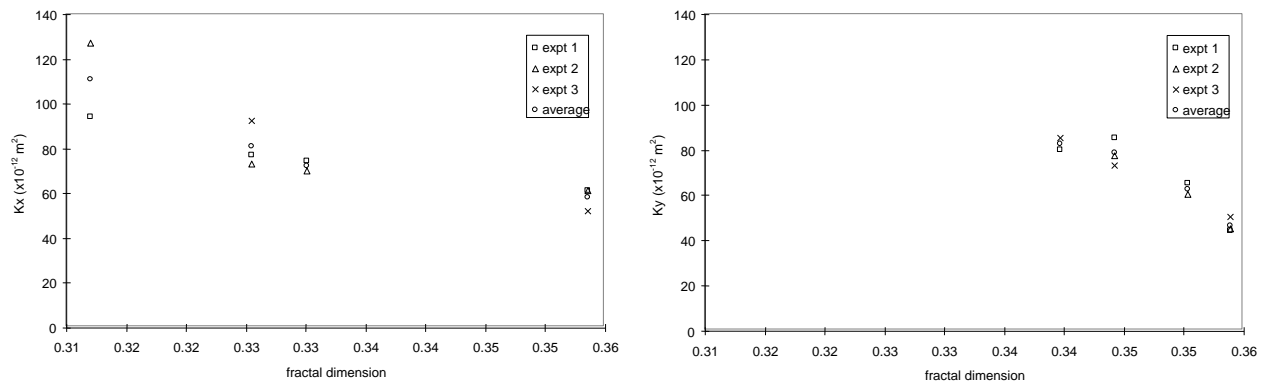


Figure 10:

Permeability plotted against fractal dimension for weft (left) and warp (right) new concept Carr fabrics

The mechanical properties of the new concept Carr fabrics are very similar and show very low scatter for all directions without FET: both warp and weft in the base fabric and the warp direction in FET fabrics (Table 4). The higher scatter in the tensile strength is attributed to the alignment of the mechanical grips in the screw-driven machine being less accurate than that of the hydraulic grips in the servo-hydraulic machine.

Table 4: Mechanical properties of new concept Carr fabrics

| Secant moduli (GPa) | | | | |
|---------------------|--------------------|-------------|----------------|-------------|
| | <i>Compression</i> | | <i>Tension</i> | |
| | <i>Warp</i> | <i>Weft</i> | <i>Warp</i> | <i>Weft</i> |
| A | 47 ± 1.10% | 40 ± 0.75% | 49 ± 0.89% | 42 ± 0.37% |
| B | 48 ± 1.02% | 43 ± 0.58% | 49 ± 1.34% | 45 ± 0.82% |
| C | 47 ± 1.24% | 44 ± 0.38% | 49 ± 0.70% | 45 ± 1.25% |
| D | 48 ± 0.51% | 47 ± 0.44% | 50 ± 0.38% | 49 ± 0.84% |
| %SD | 0.58% | 5.28% | 0.58% | 5.70% |
| Strengths (MPa) | | | | |
| | <i>Compression</i> | | <i>Tension</i> | |
| | <i>Warp</i> | <i>Weft</i> | <i>Warp</i> | <i>Weft</i> |
| A | 315 ± 1.01% | 317 ± 0.25% | 706 ± 4.49% | 542 ± 3.41% |
| B | 317 ± 0.14% | 316 ± 0.86% | 704 ± 2.88% | 595 ± 3.51% |
| C | 317 ± 0.23% | 314 ± 1.55% | 718 ± 4.08% | 556 ± 4.97% |
| D | 317 ± 0.22% | 317 ± 0.18% | 720 ± 4.93% | 602 ± 3.44% |
| %SD | 0.24% | 0.33% | 1.01% | 4.43% |

The weft compression and weft tension moduli decrease broadly in line with the expectations from rule-of-mixtures. The weft tension strength decreases in the same sequence as the fractal dimension (Figure 11). The compression and warp tension strengths are barely affected by the presence of the FET.

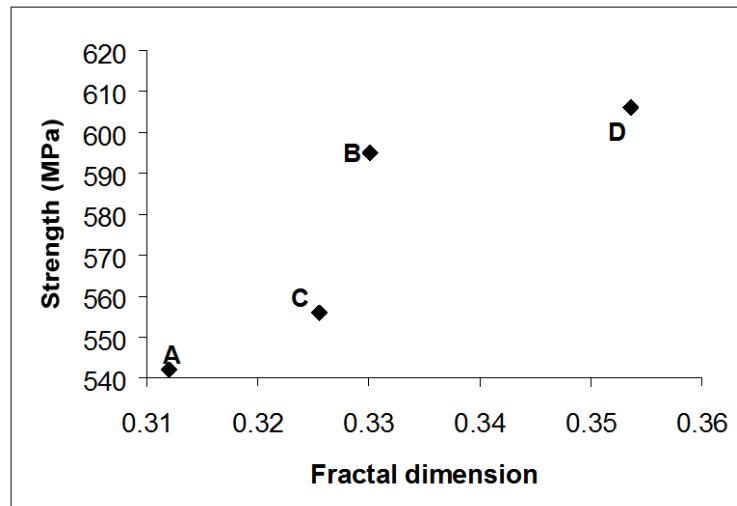


Figure 11: Weft tensile strength plotted against fractal dimension for new concept Carr fabrics

Comparison of different fabric sets

Table 5 and Figure 12 present the fractal dimensions and measured permeabilities of the Brochier and new concept Carr FET fabrics. The point at the right-hand end of the twill fabric line is for Brochier material of a lower areal weight, whilst the other points are for new concept Carr fabrics. Note that both the Brochier satin and the new concept Carr fabrics can have permeability doubled by the insertion of flow-enhancement tows.

Table 5: Fractal dimensions and permeabilities for two sets of fabric

| Fabric | Fractal dimensions | Permeability (10^{-12} m^2) |
|----------------------------------|--------------------|---|
| <i>Brochier fabrics: 290 gsm</i> | | |
| Satin (no FET) | 0.424 | 8-18 |
| Twill | 0.364 | 34-54 |
| Injectex FET | 0.356 | 19-36 |
| <i>Carr fabrics: 340-372 gsm</i> | | |
| D (base twill) | 0.354 | 41-61 |
| B (20% FET) | 0.330 | 54-75 |
| C (14% FET) | 0.326 | 67-92 |
| A (33% FET) | 0.312 | 72-126 |

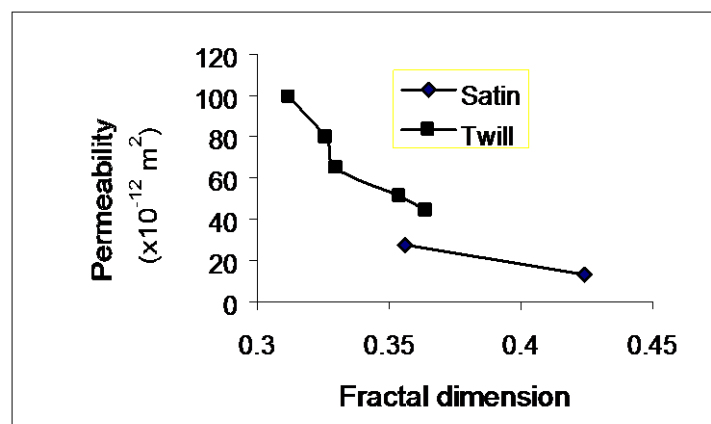


Figure 12: Permeability plotted against fractal dimension for Brochier and new concept Carr fabrics

Summary

In this paper we have considered routes to permit the microstructural features of woven composites to be quantified. These techniques have been applied to real woven reinforcement materials. Certain processing and mechanical properties can be correlated to the quantified microstructures, even when no differences can be discerned by eye. New concept Carr fabrics have been developed which appear to achieve appropriate process-property-structure relationships for commercial application, *i.e.* flow enhancement with minimal reduction in mechanical properties.

Acknowledgements

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